

THE EFFECT OF VARIOUS FACTORS ON THE RATE OF  
DIFFUSION IN STEEPING SORGHUM GRAINS

by

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## INTRODUCTION

### Present State of Starch Industry

During the past year, the commercial feasibility of preparing starch from sorghum grains has been investigated at this college. The method of preparing starch, which is being studied, is similar to that used for preparing starch from corn (McCoy and Sjostrom, 1).

According to the present method of making starch from corn, the grain, after being steeped in warm water containing sulfurous acid, is broken up and the germ separated and washed. The rest of the material is finely ground and subjected to further separating operations with water, containing sulfurous acid, in copper reels or on copper shaking screens to remove the coarse bran and fiber particles. The liquid slurry from this coarse slop system is subjected to a series of sieving operations in silk reels or on silk shakers to remove the finer particles of bran and fiber. The slurry, containing starch and gluten, which passes through the silk sieves, is run over starch tables on which the starch

settles and from which the water and gluten tails off into gluten settlers. The starch on the tables is removed therefrom by flushing, and is passed in a diluted condition onto filters, on which the starch is washed with fresh water; this operation removed from the starch the soluble impurities. The moist starch is then carefully dried at low temperature.

Gulrajany (2) proposed a method similar to that described above, to prepare starch from jowar, which is a type of millet raised in India. Newkirk (3) reported that the Corn Products Refining Company has used kafir corn as a raw material for starch production. Newkirk<sup>1</sup> stated that the major problem remaining to be solved in the use of sorghum grains as a raw material for starch production, is to find a method of getting a clean separation of the germ from the endosperm.

The steeping of the corn is described by Moffett (4) as follows:

The corn is steeped for 30 to 40 hours with water containing 0.25 per cent to 0.35 per cent of sulfur dioxide. The sulfur dioxide prevents the formation of mildew and slime in the steep.

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<sup>1</sup> Personal communication from Mr. L. J. Newkirk, Chicago, Ill.



By means of steam siphons, the water is kept in circulation and at the same time heated and maintained at a temperature varying from 115°F. to 125°F. The temperature must be sufficiently high to effect a rapid softening of the corn and yet not be so high that the starch granules will be weakened by hydrolysis or undue enzymic action.

The customary type of steeping apparatus is described by Curry and Sayre (5):

It has been customary to steep corn with dilute sulfurous acid in a counter-current diffusion battery consisting of a series of tanks through which the steeping liquid is circulated in such a manner that the fresh liquid comes in contact first with the oldest batch of corn and then passes through the next oldest batch and so on, and is ultimately withdrawn from the system after passing through the freshest batch of corn; .....

In a few words, the grain is steeped to soften it, to facilitate later operations, and to remove as much of the water-soluble material from the grain as possible (Moffett, 6). The sulfur dioxide is used to prevent bacterial action and to cause the bran to separate more readily from the endosperm in later operations.

### Theoretical Background

The program of research in manufacturing starch at the Kansas State College of Agriculture and Applied Science includes a study of all phases of the wet milling

process as applied to sorghum grains. In previous work on this project, it was found that the rate of diffusion of solubles from kafir into steep water was negligible when there was no agitation of the steep water. The work of Moffett (4, 6) and McCoy and Sjostrom (1) indicated that the steep water was recirculated within each steep tank. It was not stated whether this was done to increase the rate of diffusion. Curry and Sayre (5) indicated that the forward flow of steep water was sufficient agitation to give a good rate of diffusion. On a recent visit to a plant of the Corn Products Refining Company, the writer found that the plant does not practice recirculation of water in the steeping vats. It was desired to find a relationship between the rate of flow of steep water and the rate of diffusion of solubles. This knowledge is necessary to determine whether it is desirable to recirculate steep water in a commercial steeping battery; and, if not, whether it is necessary to recirculate steep water in a small pilot-plant steeping battery, in order to simulate commercial plant conditions.

Analysis of the diffusion process by the methods of Sherwood (7) would regard the mechanism of diffusion as

being the passage of material out through the grain, and through a film of steep water which adheres to the surface of the kernel. This layer of fluid has a greater concentration of solubles than has the main body of the liquid. The resistance in the grain to the passage of material, depends only upon the temperature and the presence of chemical agents which alter its nature. The thickness, and hence the resistance, of the liquid film depends on the rate of flow of fluid. If the resistance in the grain is large compared to the resistance of the fluid film, a change in the rate of flow of fluid will not greatly affect the rate of diffusion. Although it is impossible to measure these resistances separately, the resistance of the grain may be assumed as equal to the total resistance to diffusion when the velocity of the steep water is so great that an increase in velocity does not give an increase in the rate of diffusion.

Previous work in the Chemical Laboratory of the Kansas State College of Agriculture and Applied Science (unpublished) indicated that the following equation holds fairly well:

$$\frac{ds}{d\theta} = k C_g - C_w$$

where:

$\frac{ds}{d\theta}$  is the instantaneous rate of diffusion.

$k$  is a constant which depends on factors which affect the resistance of the grain and of the fluid film to the diffusion of solubles (temperature, rate of flow of steep water, chemical agents, etc.).

$C_g$  is the concentration of soluble solids in the grain, expressed as grams per gram of moisture in the grain.

$C_w$  is the concentration of soluble solids in the steep water, expressed as grams per gram of water.

It was also determined in previous work, that 9.0 per cent of the dry weight of the Standard Blackhull kafir, which was used in this experiment, represented the total amount of Material soluble in 0.2 per cent sulfur dioxide solution, at 120°F.

## EXPERIMENTAL WORK

### Apparatus

In order to determine the effect of the rate of flow of steep water on the rate of diffusion, a single steeping cell was set up in such a way that the rate of flow of steep water could be varied at will. A schematic

diagram of the apparatus is shown in Fig. 1. The body of the steeping cell (b) was a pyrex glass pipe, three inches in diameter. The glass resisted corrosion and allowed the grain to be inspected. A gear-pump (k) circulated the steep water through the cell. An orifice (e) and manometer (d) were provided to measure the flow of steep water. A valve (g) and a by-pass (f) on the pump provided a means of controlling the flow of steep water. A perforated plate (c) in the bottom of the cell provided even distribution of fluid, and the upper end of the fluid return tube (j) was covered with a screen (i) to keep the grain out of the pump. A manometer (not shown) was provided to measure the pressure drop through the column of grain. The whole apparatus was enclosed in a constant temperature cabinet. Figures 2, 3, 4, and 5 are photographs of this apparatus. The apparatus will also be used to determine the effect of temperature and of various chemical agents on the rate of diffusion.

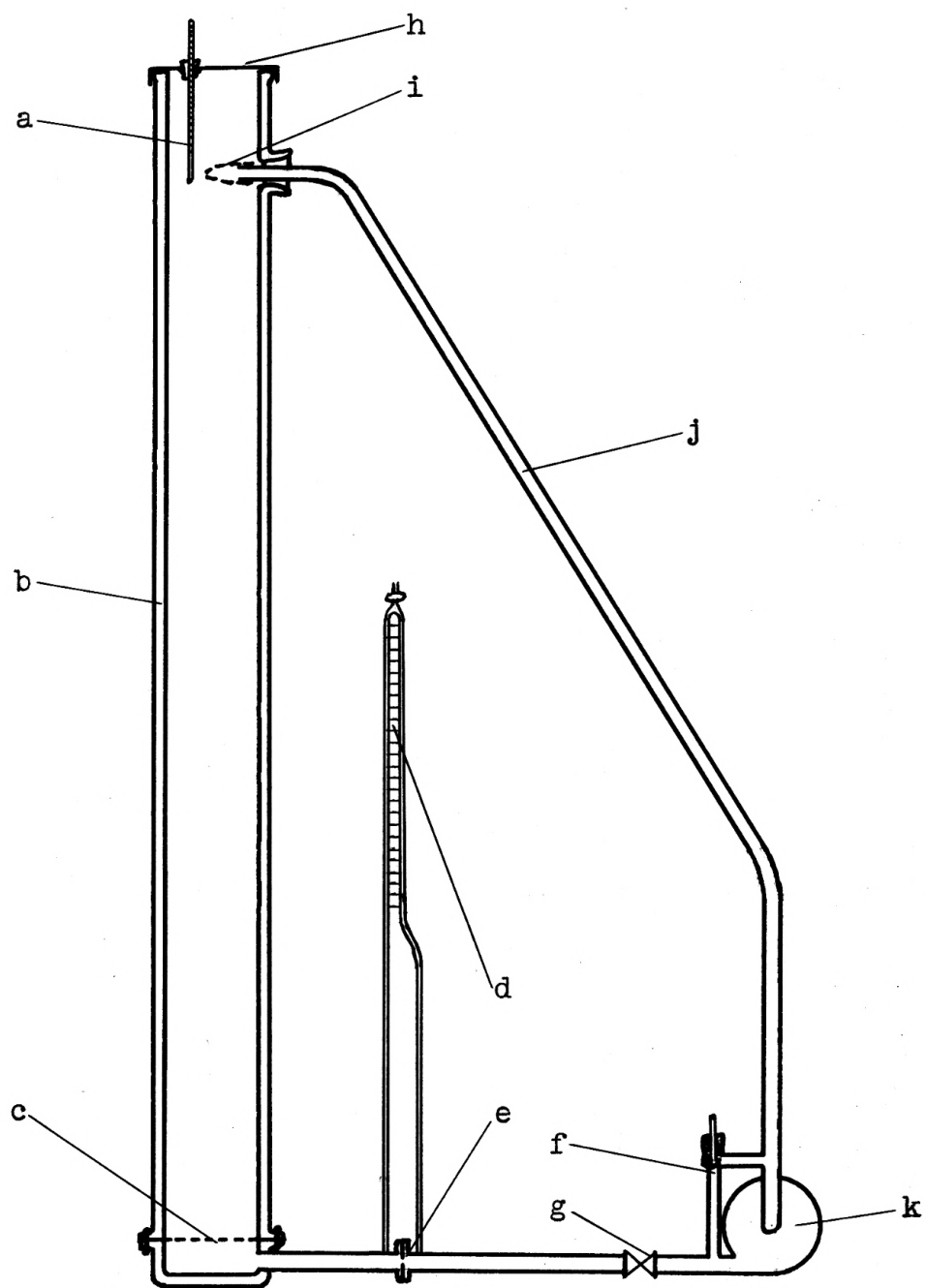


Fig. 1. Diagram of steeping apparatus.

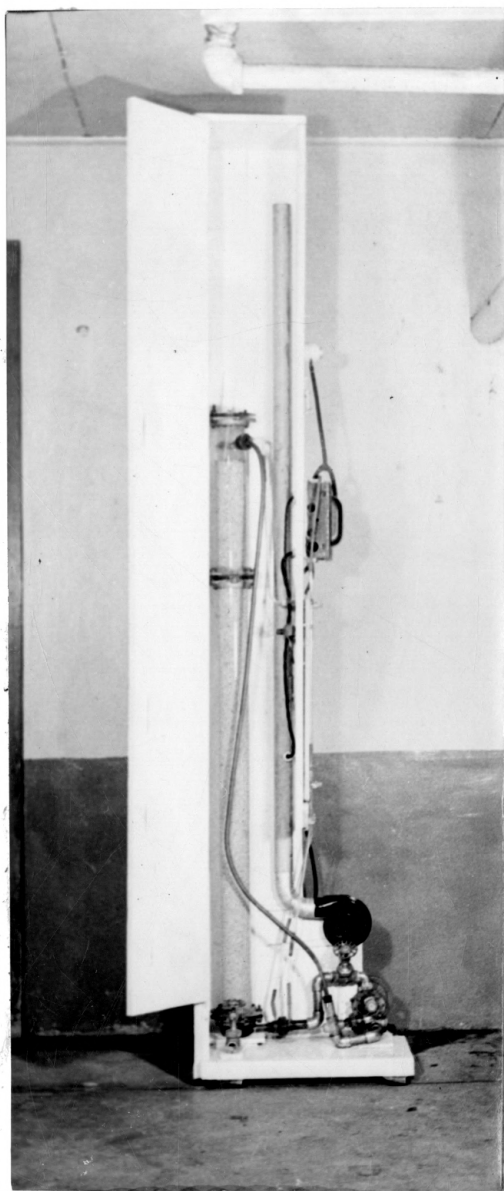


Fig. 2. Steeping apparatus, cabinet open.



Fig. 3. Steeping apparatus, cabinet closed.

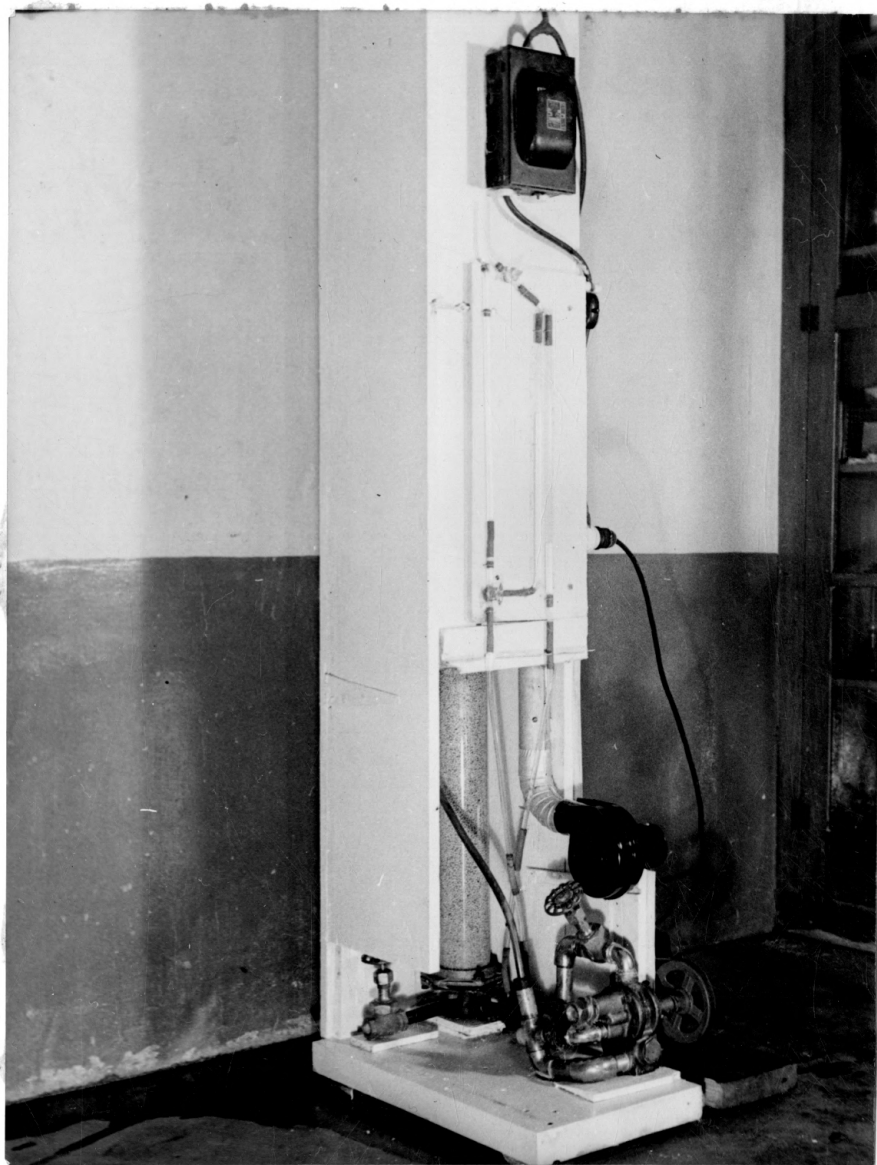


Fig. 4. Steeping apparatus, control devices.





Fig. 5. Steeping apparatus, circulating pump and orifice.

### Operation, Data and Calculations

In the operation of this apparatus, all factors were held constant during a single run. Samples were taken at specific intervals, for the purpose of determining the moisture content of the grain and the concentration of solids dissolved in the steep water. By means of a material balance, the amount of solids extracted was determined for different periods of time. Table 1 gives the experimental data from several runs using various rates of fluid flow, from 800 gallons per hour per square foot of cross section perpendicular to the direction of flow, to 11 gallons per hour per square foot. Table 2 gives data which was calculated from the data of Table 1, in the following manner:

Initial values:

dry grain as charged -  $2730 \times 0.89 = 2430$  grams

soluble solids in grain as charged -  
 $2430 \times 0.09 = 219$  grams

moisture in grain as charged -  
 $2730 \times 0.11 = 300$  grams

steep water added is 3750 grams

total water charged -  $3750 + 300 = 4050$  grams

At the end of one hour; fluid flow, 800 gallons per hour per square foot:

$$\text{moisture in grain} - 2400^2 \times \frac{26.3}{100 - 26.3} = 860 \text{ grams}$$

$$\text{grams water per gram of dry grain} - \frac{860}{2400} = 0.355$$

moisture in steep water -  $4050 - 860 = 3190$  grams,  
which is equivalent to 3.19 liters of steep water

$$\text{solubles extracted} - 3.19 \times \frac{1000}{25} \times 0.099 = 12.6 \text{ grams}$$

$$\text{solubles left in grain} - 219 - 12.6 = 203 \text{ grams}$$

At the end of three hours; fluid flow, 800 gallons per hour per square foot:

Moisture in grain, grams of water per gram of dry grain, volume of steep water, and solubles left in grain were calculated as indicated above.

Soluble solids extracted were calculated as above, plus the amount of solids removed in previous analyses, thus:

$$2.83 \times \frac{1000}{25} \times 0.191 + 2 \times 0.099 = 21.9 \text{ grams}$$

Further data were calculated in the manner indicated above.

The last five columns of Table 2 were calculated as follows:

$C_g$  is the concentration of soluble solids in the grain, expressed as grams per gram of moisture

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<sup>2</sup>The average weight of solids in the grain, after the extracted solids and moisture samples were deducted, was estimated as being 2400 grams.

in the grain -

$$\frac{203}{860} = 0.236$$

$C_w$  is the concentration of soluble solids in the steep water, expressed as grams per gram of water -

$$\frac{0.099}{25} = 0.004$$

$s$  is the grams of solids extracted per gram of dry grain charged -

$$\frac{12.6}{2430} = 0.0052$$

$\frac{ds}{d\theta}$ , the instantaneous rate of change of  $s$  with reference to time, was measured from the graph of  $s$  versus  $\theta$  (Fig. 6).

$k$ , the diffusion coefficient, was calculated from the formula -

$$\frac{ds}{d\theta} = k \quad C - C$$

Table 1. Experimental data.

| Flow of fluid,<br>gal./sq.<br>per hour | Total time<br>in cell<br>hours | Moisture in<br>grain,<br>per cent |      | Dissolved solids<br>grams/25 ml.<br>of steep water |       |
|--|--------------------------------|-----------------------------------|------|--|-------|
| All rates                              | 0                              | 11.0                              |      | 0.0  |       |
| 800                                    | 1                              | 26.7                              | 25.8 | 0.100  | 0.098 |
|  | 3                              | 33.5                              | 33.5 | 0.191  | 0.191 |
|  | 6                              | 36.3                              | 39.0 | 0.297  | 0.304 |
|  | 10                             | 39.6                              | 40.0 | 0.442  | 0.449 |
|  | 16                             | 36.8                              | 39.8 | 0.533  | 0.529 |
|  | 22                             | 40.1                              | 40.2 | 0.566  | 0.576 |
|  | 28                             | 40.7                              | 40.2 | 0.578  | 0.573 |
|  | 34                             | 40.8                              | 40.8 | 0.582  | 0.590 |
|  | 40                             | 40.2                              | 39.2 | 0.622  | 0.626 |
|  | 46                             | 40.0                              | 39.8 | 0.643  | 0.653 |
| 310                                    | 1                              | 26.3                              | 26.3 | 0.102  | 0.101 |
|  | 3                              | 34.3                              | 34.3 | 0.201  | 0.204 |
|  | 6                              | 39.1                              | 38.9 | 0.365  | 0.361 |
|  | 10                             | 39.1                              | 39.6 | 0.365  | 0.361 |
|  | 16                             | 39.7                              | 39.6 | 0.506  | 0.503 |
|  | 22                             | 40.6                              | 40.8 | 0.534  | 0.539 |
|  | 28                             | 40.3                              | 40.4 | 0.562  | 0.574 |
|  | 34                             | 40.5                              | 40.1 | 0.552  | 0.554 |
|  | 40                             | 40.5                              | 40.7 | 0.552  | 0.551 |
|  | 46                             | 40.7                              | 40.5 | 0.581  | 0.591 |
| 170                                    | 1                              | 27.3                              | 27.7 | 0.109  | 0.109 |
|  | 3                              | 35.3                              | 35.5 | 0.216  | 0.214 |
|  | 6                              | 39.2                              | 38.9 | 0.376  | 0.360 |
|  | 10                             | 40.3                              | 40.6 | 0.486  | 0.494 |
|  | 16                             | 39.8                              | 39.9 | 0.565  | 0.571 |
|  | 22                             | 39.7                              | 39.9 | 0.565  | 0.547 |
|  | 28                             | 40.0                              | 40.1 | 0.599  | 0.594 |
|  | 34                             | 40.0                              | 40.3 | 0.579  | 0.574 |
|  | 40                             | 40.7                              | 40.6 | 0.581  | 0.577 |
|  | 46                             | 40.3                              | 40.0 | 0.586  | 0.593 |

All runs use six pounds (2730 grams) of Standard Blackhull kafir (11.0 per cent moisture) and one gallon (3.75 liters) of 0.2 per cent sulfur dioxide solution. Temperature in all cases was 120° F. ( $\pm 3^\circ$  F.). All samples were taken in duplicate.

Table 1. (Cont.)

| Flow of fluid,<br>gal./sq. ft.<br>per hour | Total time<br>in cell<br>hours | Moisture in<br>grain,<br>per cent | Dissolved solids<br>grams/25 ml.<br>of steep water |       |       |
|--|--------------------------------|-----------------------------------|--|-------|-------|
| All rates                                  | 0                              | 11.0                              | 0.0  |       |       |
| 54   | 1                              | 25.7                              | 25.9   | 0.120 | 0.122 |
|  | 3                              | 32.6                              | 32.4   | 0.177 | 0.173 |
|  | 6                              | 36.7                              | 36.9   | 0.285 | 0.296 |
|  | 10                             | 38.6                              | 39.0   | 0.432 | 0.428 |
|  | 16                             | 39.8                              | 39.4   | 0.577 | 0.577 |
|  | 22                             | 40.4                              | 40.1   | 0.639 | 0.634 |
|  | 28                             | 40.0                              | 40.2   | 0.633 | 0.641 |
|  | 34                             | 41.2                              | 40.5   | 0.666 | 0.653 |
|  | 40                             | 40.8                              | 40.4   | 0.700 | 0.701 |
|  | 46                             | 40.3                              | 40.3   | 0.702 | 0.709 |
| 28   | 1                              | 27.4                              | 27.3   | 0.093 | 0.095 |
|  | 3                              | 32.4                              | 32.4   | 0.148 | 0.153 |
|  | 6                              | 36.4                              | 36.4   | 0.229 | 0.232 |
|  | 10                             | 38.6                              | 38.6   | 0.336 | 0.350 |
|  | 16                             | 39.9                              | 39.0   | 0.504 | 0.500 |
|  | 22                             | 39.5                              | 39.6   | 0.635 | 0.619 |
|  | 28                             | 39.8                              | 39.8   | 0.651 | 0.661 |
|  | 36                             | 39.7                              | 39.6   | 0.683 | 0.679 |
|  | 44                             | 39.7                              | 39.9   | 0.724 | 0.748 |
| 24   | 1                              | 25.7                              | 26.1   | 0.113 | 0.041 |
|  | 3                              | 31.1                              | 31.4   | 0.159 | 0.155 |
|  | 6                              | 35.3                              | 35.3   | 0.208 | 0.203 |
|  | 10                             | 38.4                              | 38.5   | 0.328 | 0.327 |
|  | 16                             | 39.8                              | 39.8   | 0.481 | 0.486 |
|  | 22                             | 38.9                              | 39.4   | 0.613 | 0.616 |
|  | 28                             | 39.4                              | 39.8   | 0.659 | 0.677 |
|  | 36                             | 39.9                              | 40.2   | 0.696 | 0.690 |
|  | 44                             | 40.2                              | 40.0   | 0.712 | 0.720 |
| 11   | 1                              | 27.5                              | 27.6   | 0.038 | 0.029 |
|  | 3                              | 35.1                              | 35.3   | 0.033 | 0.032 |
|  | 6                              | 38.9                              | 39.8   | 0.108 | 0.116 |
|  | 10                             | 39.6                              | 39.6   | 0.119 | 0.123 |
|  | 16                             | 39.9                              | 40.9   | 0.526 | 0.528 |
|  | 22                             | 40.0                              | 39.8   | 0.599 | 0.586 |
|  | 28                             | 40.0                              | 40.7   | 0.636 | 0.623 |
|  | 34                             | 40.3                              | 38.3   | 0.607 | 0.617 |
|  | 40                             | 40.2                              | 40.7   | 0.620 | 0.602 |
|  | 46                             | 39.7                              | 39.8   | 0.631 | 0.631 |

Table 2. Data calculated from Table 1.

| Fluid<br>flow,<br>gal./hr.: | Time,<br>hours: | Water in:<br>grain,<br>grams | Gr. water:<br>per gr.<br>dry grain: | Steep<br>water,<br>liters: | Solubles, grams:<br>in<br>extracted: | grams:<br>in<br>grain: | $C_g$ | $C_w$ | $s$<br>$\times 10^3$ | $\frac{ds}{d\theta}$<br>$\times 10^3$ | $k$<br>$\times 10^3$ |
|-----------------------------|-----------------|------------------------------|-------------------------------------|----------------------------|--------------------------------------|------------------------|-------|-------|----------------------|---------------------------------------|----------------------|
| All                         | 0               | 300                          | 0.124                               | 3.75                       | 0.0                                  | 219                    | 0.730 | 0     | 0                    |                                       |                      |
| 800                         | 1               | 860                          | 0.355                               | 3.75                       | 12.6                                 | 203                    | 0.236 | 0.004 | 5.2                  | 2.6                                   | 11.2                 |
|                             | 3               | 1220                         | 0.502                               | 2.83                       | 21.9                                 | 197                    | 0.161 | 0.008 | 9.0                  | 1.8                                   | 11.8                 |
|                             | 6               | 1460                         | 0.600                               | 2.59                       | 31.7                                 | 187                    | 0.128 | 0.012 | 13.1                 | 1.4                                   | 12.0                 |
|                             | 10              | 1580                         | 0.650                               | 2.47                       | 45.2                                 | 174                    | 0.110 | 0.018 | 18.6                 | 1.0                                   | 10.9                 |
|                             | 16              | 1570                         | 0.646                               | 2.48                       | 54.8                                 | 164                    | 0.104 | 0.021 | 22.6                 | 0.5                                   | 6.4                  |
|                             | 22              | 1590                         | 0.655                               | 2.46                       | 59.6                                 | 159                    | 0.100 | 0.023 | 24.5                 | 0.23                                  | 3.0                  |
|                             | 28              | 1600                         | 0.659                               | 2.45                       | 61.1                                 | 158                    | 0.099 | 0.023 | 25.2                 | 0.12                                  | 1.6                  |
| 310                         | 1               | 850                          | 0.354                               | 3.20                       | 13.0                                 | 206                    | 0.242 | 0.004 | 5.4                  | 2.9                                   | 12.2                 |
|                             | 3               | 1260                         | 0.525                               | 2.79                       | 22.8                                 | 191                    | 0.152 | 0.008 | 9.5                  | 2.1                                   | 14.6                 |
|                             | 6               | 1530                         | 0.640                               | 2.52                       | 37.2                                 | 182                    | 0.119 | 0.015 | 15.5                 | 1.6                                   | 15.4                 |
|                             | 10              | 1560                         | 0.650                               | 2.49                       | 47.9                                 | 171                    | 0.110 | 0.019 | 19.9                 | 0.7                                   | 7.8                  |
|                             | 16              | 1580                         | 0.657                               | 2.47                       | 52.0                                 | 167                    | 0.106 | 0.020 | 21.7                 | 0.33                                  | 3.9                  |
|                             | 22              | 1650                         | 0.685                               | 2.40                       | 54.8                                 | 164                    | 0.099 | 0.021 | 22.8                 | 0.17                                  | 2.2                  |
|                             | 28              | 1620                         | 0.675                               | 2.43                       | 59.6                                 | 160                    | 0.099 | 0.023 | 24.4                 | 0.16                                  | 2.1                  |



Table 2. (Cont.)

| Fluid<br>flow,<br>gal./hr.: | Time,<br>hours: | Water in:<br>grain,<br>grams: | Gr. water:<br>per gr.<br>dry grain: | Steep<br>water,<br>liters: | Solubles, grams:<br>in<br>extracted grain: | $C_g$ | $C_w$ | $s$<br>$\times 10^3$ | $\frac{ds}{d\theta}$<br>$\times 10^3$ | $k$<br>$\times 10^3$ |
|-----------------------------|-----------------|-------------------------------|-------------------------------------|----------------------------|--|-------|-------|----------------------|---------------------------------------|----------------------|
| All                         | 0               | 300                           | 0.124                               | 3.75                       | 0.0  | 219   | 0.730 | 0                    | 0                                     |                      |
| 170                         | 1               | 910                           | 0.380                               | 3.14                       | 13.7                                       | 205   | 0.226 | 0.004                | 5.7                                   | 3.3                  |
|                             | 3               | 1310                          | 0.578                               | 2.74                       | 23.8                                       | 195   | 0.149 | 0.009                | 9.9                                   | 2.7                  |
|                             | 6               | 1540                          | 0.641                               | 2.51                       | 37.6                                       | 181   | 0.118 | 0.015                | 15.7                                  | 1.8                  |
|                             | 10              | 1630                          | 0.679                               | 2.42                       | 48.8                                       | 170   | 0.104 | 0.020                | 20.3                                  | 0.9                  |
|                             | 16              | 1590                          | 0.662                               | 2.46                       | 58.3                                       | 161   | 0.102 | 0.023                | 24.3                                  | 0.5                  |
|                             | 22              | 1590                          | 0.662                               | 2.46                       | 58.3                                       | 161   | 0.101 | 0.022                | 24.3                                  | 0.2                  |
|                             | 28              | 1610                          | 0.668                               | 2.44                       | 62.8                                       | 156   | 0.097 | 0.024                | 26.2                                  | 0.15                 |
| 54                          | 1               | 830                           | 0.348                               | 3.22                       | 15.5                                       | 203   | 0.245 | 0.005                | 6.5                                   | 2.3                  |
|                             | 3               | 1160                          | 0.482                               | 2.89                       | 20.4                                       | 199   | 0.172 | 0.007                | 8.5                                   | 1.4                  |
|                             | 6               | 1400                          | 0.582                               | 2.65                       | 31.3                                       | 188   | 0.135 | 0.012                | 13.0                                  | 1.3                  |
|                             | 10              | 1520                          | 0.634                               | 2.53                       | 44.7                                       | 174   | 0.114 | 0.017                | 18.6                                  | 1.3                  |
|                             | 16              | 1570                          | 0.656                               | 2.48                       | 59.2                                       | 160   | 0.102 | 0.023                | 24.7                                  | 0.7                  |
|                             | 22              | 1620                          | 0.674                               | 2.43                       | 65.1                                       | 154   | 0.095 | 0.025                | 27.1                                  | 0.24                 |
|                             | 28              | 1600                          | 0.669                               | 2.45                       | 67.1                                       | 152   | 0.095 | 0.025                | 28.0                                  | 0.12                 |
| 28                          | 1               | 870                           | 0.348                               | 3.18                       | 12.0                                       | 207   | 0.238 | 0.004                | 5.0                                   | 2.0                  |
|                             | 3               | 1150                          | 0.479                               | 2.90                       | 17.6                                       | 201   | 0.175 | 0.006                | 7.3                                   | 1.2                  |
|                             | 6               | 1360                          | 0.572                               | 2.69                       | 25.2                                       | 194   | 0.143 | 0.009                | 10.5                                  | 1.1                  |
|                             | 10              | 1510                          | 0.624                               | 2.54                       | 35.8                                       | 183   | 0.121 | 0.014                | 14.9                                  | 1.1                  |
|                             | 16              | 1570                          | 0.653                               | 2.48                       | 51.1                                       | 168   | 0.107 | 0.020                | 21.3                                  | 1.0                  |
|                             | 22              | 1570                          | 0.653                               | 2.48                       | 64.5                                       | 154   | 0.098 | 0.025                | 26.9                                  | 0.7                  |
|                             | 28              | 1590                          | 0.662                               | 2.46                       | 68.2                                       | 151   | 0.095 | 0.026                | 28.4                                  | 0.3                  |



Table 2. (Cont.)

| Fluid<br>flow,<br>gal./hr.: | Time,<br>hours: | Water in:<br>grain,<br>grams | Gr. water:<br>per gr.<br>dry grain: | Steep<br>water,<br>liters: | Solubles, grams:<br>in :<br>extracted: | $C_g$ | $C_w$ | $s$<br>$\times 10^3$ | $\frac{ds}{dC}$<br>$\times 10^3$ | $k$<br>$\times 10^3$ |
|-----------------------------|-----------------|------------------------------|-------------------------------------|----------------------------|--|-------|-------|----------------------|----------------------------------|----------------------|
| All                         | 0               | 300                          | 0.124                               | 3.75                       | 0.0                                    | 219   | 0.730 | 0                    | 0                                |                      |
| 24                          |                 | 1340                         | 0.350                               | 3.21                       | 14.4                                   | 205   | 0.244 | 0.004                | 6.0                              | 2.1                  |
|                             |                 | 1090                         | 0.440                               | 2.96                       | 18.8                                   | 200   | 0.183 | 0.006                | 7.8                              | 1.5                  |
|                             |                 | 1310                         | 0.546                               | 2.74                       | 23.0                                   | 196   | 0.150 | 0.008                | 9.6                              | 1.1                  |
|                             | 10              | 1500                         | 0.624                               | 2.55                       | 34.3                                   | 185   | 0.123 | 0.013                | 13.9                             | 0.9                  |
|                             | 16              | 1590                         | 0.662                               | 2.46                       | 50.2                                   | 169   | 0.106 | 0.020                | 20.9                             | 0.8                  |
|                             | 22              | 1520                         | 0.630                               | 2.53                       | 64.8                                   | 154   | 0.101 | 0.025                | 27.0                             | 0.7                  |
|                             | 28              | 1570                         | 0.656                               | 2.48                       | 70.1                                   | 149   | 0.095 | 0.027                | 29.2                             | 0.4                  |
|                             | 1               | 915                          | 0.381                               | 3.13                       | 4.3                                    | 215   | 0.235 | 0.001                | 1.8                              | *                    |
|                             | 3               | 1300                         | 0.543                               | 2.75                       | 3.7                                    | 215   | 0.165 | 0.001                | 1.5                              | *                    |
|                             | 6               | 1560                         | 0.650                               | 2.49                       | 11.3                                   | 208   | 0.133 | 0.004                | 4.7                              |                      |
|                             | 10              | 1570                         | 0.656                               | 2.48                       | 12.4                                   | 207   | 0.132 | 0.005                | 5.2                              |                      |
|                             | 16              | 1630                         | 0.678                               | 2.42                       | 51.6                                   | 167   | 0.102 | 0.021                | 21.5                             |                      |
|                             | 22              | 1590                         | 0.664                               | 2.46                       | 59.9                                   | 159   | 0.100 | 0.024                | 25.0                             |                      |
|                             | 28              | 1620                         | 0.677                               | 2.43                       | 64.0                                   | 155   | 0.096 | 0.025                | 26.7                             |                      |

\*The samples from this run were not representative, due to insufficient mixing.

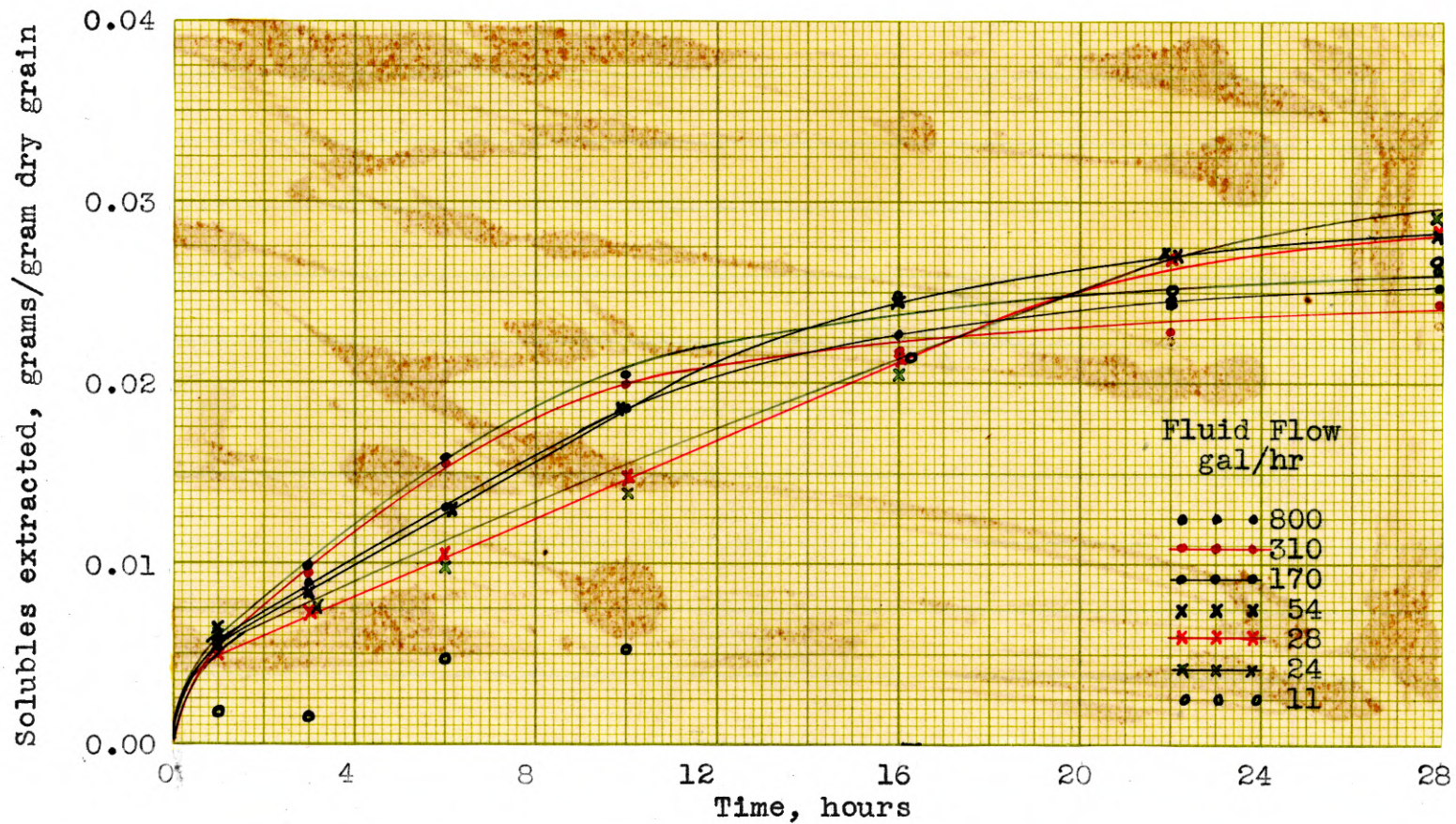


Fig. 6. Solubles extracted versus time of steeping.

Table 3. Diffusion coefficient versus flow of steep water.

| Rate of flow:<br>of<br>steep water | Average<br>: diffusion<br>: coefficient | Reciprocal<br>: of<br>: rate of flow | Reciprocal<br>: of diffusion<br>: coefficient |
|------------------------------------|---|--------------------------------------|---|
| 800                                | 0.0105                                  | 0.0012                               | 95  |
| 310                                | 0.0108                                  | 0.0032                               | 93  |
| 170                                | 0.0138                                  | 0.0059                               | 73  |
| 54                                 | 0.0102                                  | 0.0185                               | 98  |
| 28                                 | 0.0091                                  | 0.0358                               | 104   |
| 24                                 | 0.0085                                  | 0.0417                               | 118   |
| 11                                 | ?                                       | 0.0909                               | ?   |

The diffusion coefficients for each run, calculated at 1, 3, 6, 10, and 16 hours, were averaged and compared in Table 3. In Figure 7, the reciprocal of the coefficient, or the resistance to diffusion, is plotted against the reciprocal of the rate of flow. Figure 8 shows the variation of moisture content of the grain with time.

In order to check the variations of the diffusion coefficient, a run was made in which steep water containing an appreciable amount of soluble solids was removed

after 22.3 hours of steeping. The steeping cell was then filled with fresh sulfurous acid solution. The experimental results are given in Table 4. Table 5 contains data which were calculated from the data of Table 4 by methods previously described. Figure 9 was used in calculating the data of Table 5. Figure 10 shows the diffusion coefficient as given in Table 5, plotted against time of steeping.



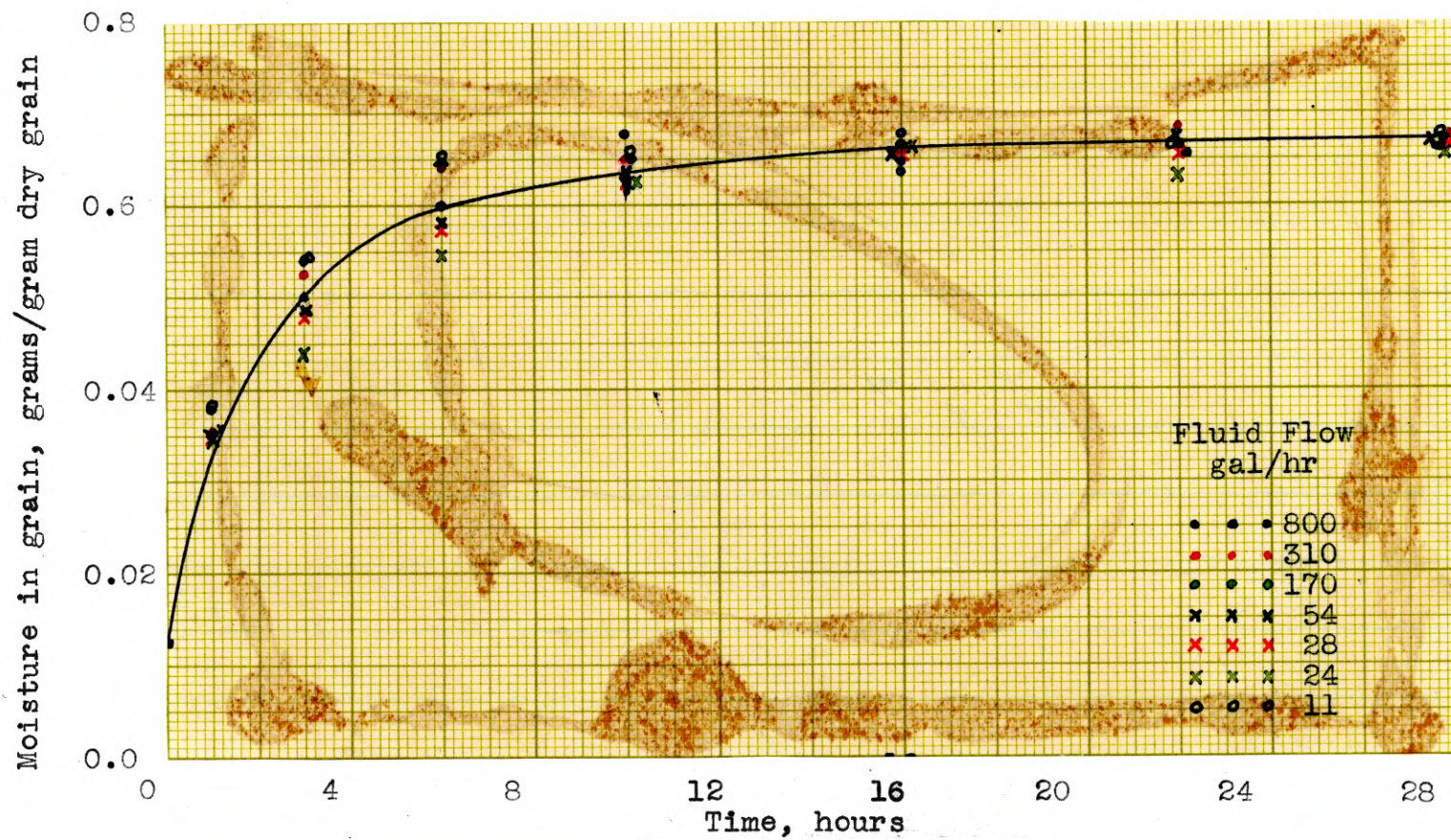


Fig. 7. Moisture content of grain versus time of steeping.



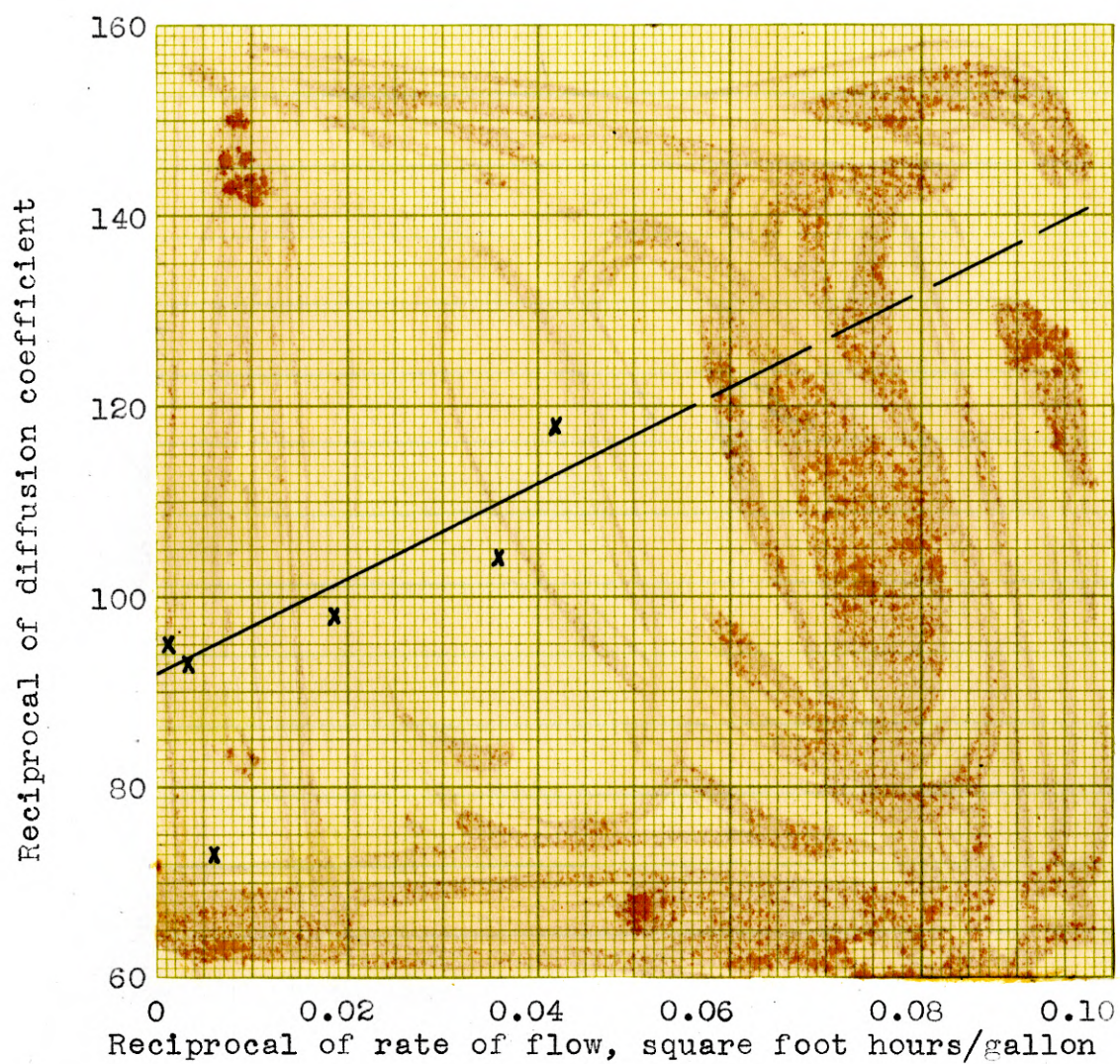


Fig. 8. Diffusion coefficient versus rate of flow of steep water.

Table 4. Experimental data, check run<sup>4</sup>.

| : Total time in cell : | Moisture in grain |      | Dissolved solids, grams/ |       |
|------------------------|-------------------|------|--------------------------|-------|
| : hours :              | per cent          |      | 25 ml. of steep water    |       |
| : 0 :                  | 11.0              |      | 0                        |       |
| 1                      | 24.2              | 27.0 | 0.114                    | 0.115 |
| 3                      | 34.2              | 34.4 | 0.230                    | 0.225 |
| 6                      | 38.6              | 38.8 | 0.364                    | 0.355 |
| 10                     | 40.0              | 39.6 | 0.444                    | 0.454 |
| 16                     | 38.6              | 38.5 | 0.581                    | 0.584 |
| 22                     | 38.7              | 38.7 | 0.608                    | 0.623 |
| **                     | **                | **   | **                       | **    |
| 23                     | 39.0              | 38.8 | 0.152                    | 0.156 |
| 24                     | 39.9              | 40.0 | 0.173                    | 0.175 |
| 25                     | 40.6              | 40.4 | 0.189                    | 0.185 |
| 27                     | 40.4              | 41.2 | 0.228                    | 0.222 |
| 30                     | 40.9              | 40.8 | 0.278                    | 0.284 |
| 34                     | 40.5              | 39.9 | 0.324                    | 0.319 |
| 40                     | 40.5              | 40.8 | 0.355                    | 0.349 |
| 46                     | 40.6              | 40.5 | 0.378                    | 0.377 |

<sup>4</sup>Six pounds (2730 grams) of Standard Blackhull kafir (11.0 per cent moisture) and one gallon (3.75 liters) of 0.2 per cent sulfur dioxide solution were used. Temperature was 120° F. Rate of flow of steep water was 300 gallons per hour per square foot. Samples were taken in duplicate.

\*\*When 22.3 hours had elapsed, steep water containing 56.8 grams of dissolved solids was removed and fresh sulfurous acid was added in its place.

Table 5. Data calculated from Table 4.

| :Time,<br>:hours: | Water in:<br>grain, | Steep<br>:water,<br>:grams | Solubles, grams:<br>:in :<br>:extracted: | grain:<br>:grain: | $C_g$ | $C_w$ | $s$<br>$\times 10^3$ | $\frac{ds}{d\theta}$<br>$\times 10^3$ | $k$<br>$\times 10^3$ |
|-------------------|---------------------|----------------------------|--|-------------------|-------|-------|----------------------|---------------------------------------|----------------------|
| 0                 | 300                 | 3.75                       | 0.0                                      | 219               | 0.730 |       |                      |                                       |                      |
| 1                 | 850                 | 3.20                       | 14.7                                     | 204               | 0.240 | 0.005 | 6.1                  | 2.9                                   | 12.1                 |
| 3                 | 1250                | 2.80                       | 25.7                                     | 193               | 0.168 | 0.009 | 10.7                 | 1.9                                   | 11.9                 |
| 6                 | 1520                | 2.53                       | 37.1                                     | 182               | 0.120 | 0.014 | 15.4                 | 1.3                                   | 12.2                 |
| 10                | 1590                | 2.46                       | 45.6                                     | 173               | 0.109 | 0.018 | 19.0                 | 0.9                                   | 9.9                  |
| 16                | 1510                | 2.54                       | 61.5                                     | 158               | 0.105 | 0.023 | 25.6                 | 0.7                                   | 8.5                  |
| 22                | 1520                | 2.53                       | 61.4                                     | 158               | 0.104 | 0.025 | 25.6                 | 0.5                                   | 6.3                  |
| **                | **                  | **                         | **                                       | **                | **    | **    | **                   | **                                    | **                   |
| 23                | 1530                | 2.52                       | 76.9                                     | 142               | 0.093 | 0.006 | 32.0                 | 0.95                                  | 10.9                 |
| 24                | 1600                | 2.45                       | 78.7                                     | 140               | 0.087 | 0.007 | 32.8                 | 0.87                                  | 10.8                 |
| 25                | 1630                | 2.42                       | 80.1                                     | 139               | 0.085 | 0.008 | 33.4                 | 0.80                                  | 10.4                 |
| 27                | 1660                | 2.39                       | 83.9                                     | 135               | 0.081 | 0.009 | 34.9                 | 0.75                                  | 10.4                 |
| 30                | 1660                | 2.39                       | 89.7                                     | 129               | 0.078 | 0.011 | 37.4                 | 0.70                                  | 10.4                 |
| 34                | 1610                | 2.44                       | 94.8                                     | 124               | 0.077 | 0.013 | 39.5                 | 0.45                                  | 7.1                  |
| 40                | 1640                | 2.41                       | 98.1                                     | 120               | 0.073 | 0.014 | 40.8                 | 0.25                                  | 4.2                  |
| 46                | 1640                | 2.41                       | 101.2                                    | 118               | 0.072 | 0.015 | 42.2                 | 0.15                                  | 2.6                  |

\*\*When 22.3 hours had elapsed, steep water containing 56.8 grams of dissolved solids was removed and fresh sulfurous acid was added in its place.



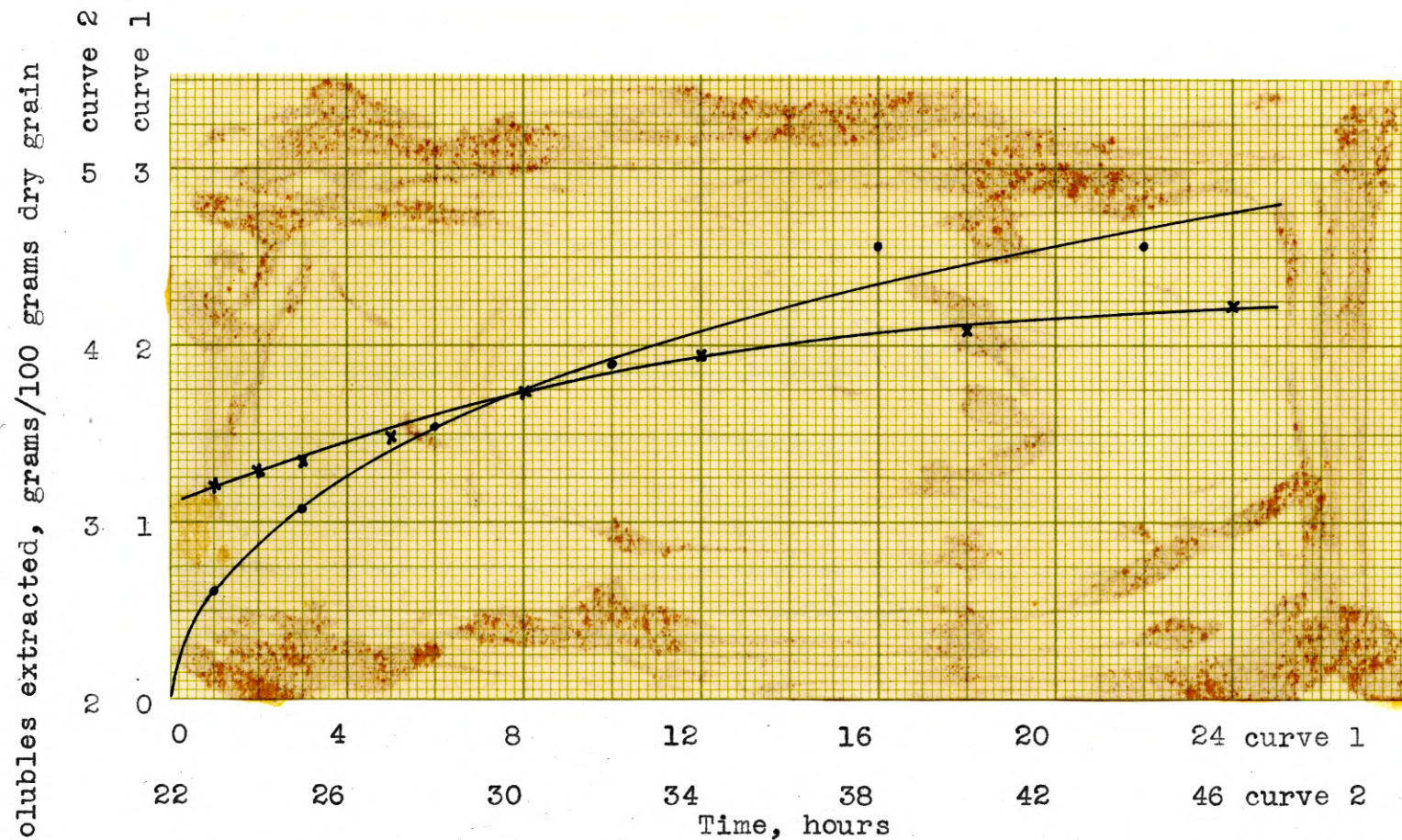


Fig. 9. Solubles extracted versus time of steeping, check run.



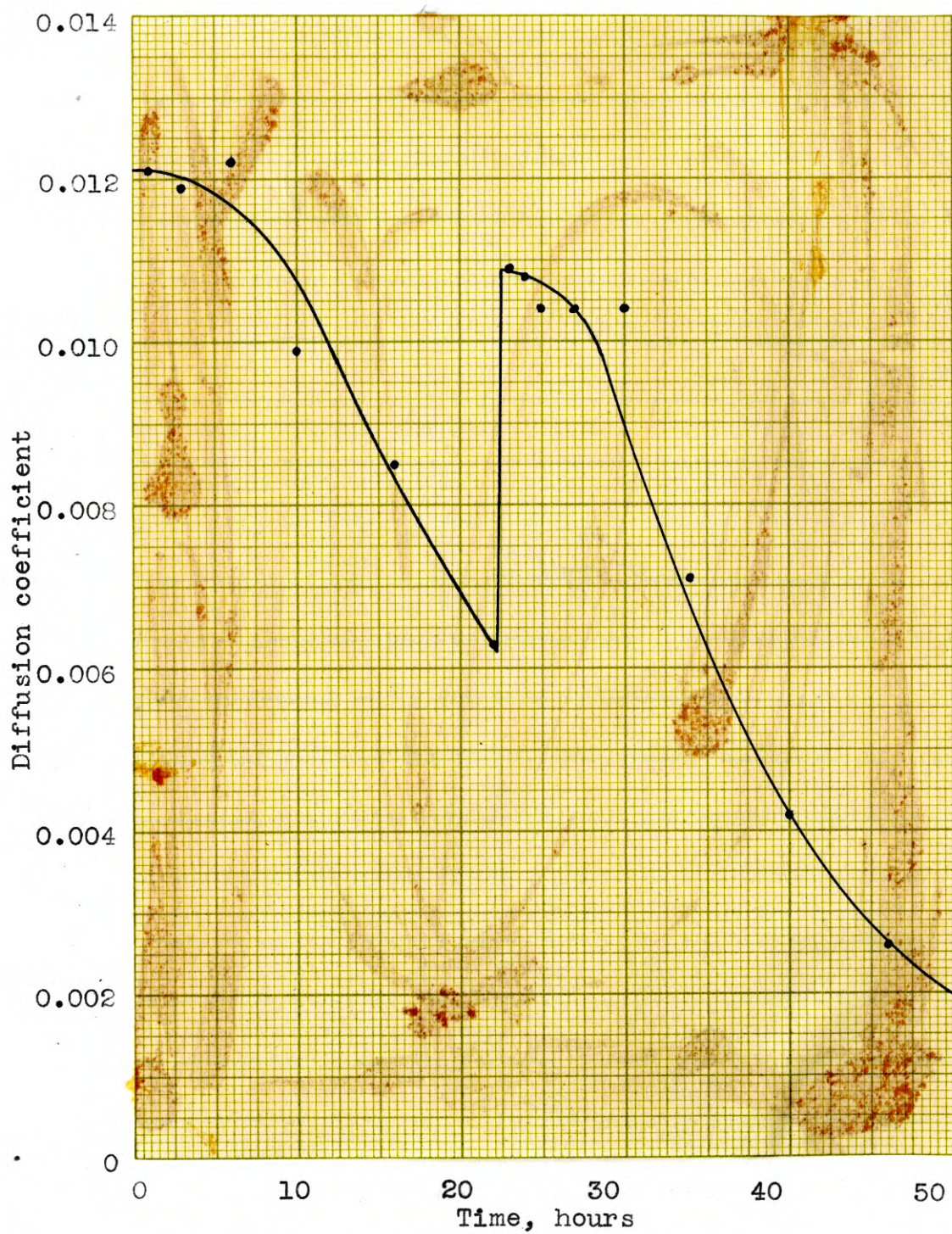


Fig. 10. Diffusion coefficient versus time of steeping.

## ANALYSIS OF DATA

### Variation of Diffusion Coefficient

By inspection of the calculated diffusion coefficients of Table 2 and Table 5, it may be seen that the diffusion coefficient decreases rapidly after about 16 hours' steeping. It may also be seen from Table 5 and Fig. 10, that the addition of fresh sulfurous acid brought the diffusion coefficient back to its original level for a period of about 10 hours.

The results of previous work showed that the diffusion coefficient changes with changing hydrogen ion concentration, and that the hydrogen ion concentration decreases during the process.

It may be expected that even with the most rigid control of conditions, the diffusion coefficient will vary during the steeping process. This is due to the fact that the solubles which are near the surface of the grain will diffuse first and will encounter less resistance to diffusion than will the solubles in the center of the grain, which diffuse later. The soluble material is dispersed throughout an inert material.

In order that the solubles will diffuse from the center to the surface of the kernel, it is necessary that there be a concentration gradient within the grain. This fact limits the assumption of an average concentration of solubles in the grain.

There is undoubtedly a change in the nature of the outer membrane of the kernel during the process of steeping. The grain increases as much as 20 per cent in volume when it becomes saturated with water. This change in space arrangement of the inert material must have some effect on the ease with which the soluble materials diffuse out. The effect of hydrogen ion concentration on the diffusion coefficient is probably due to an accompanying change in the nature of the membrane which resists diffusion.

#### Rate of Flow of Steep Water

In the wet milling of corn, it was found that ten to twelve gallons of water per bushel of corn should be used in the steeping battery (Kelling, 8). A commercial sized steeping vat, which is thirty feet deep, will hold twenty bushels of grain per square foot of



cross sectional area, when allowances are made for the swelling of the grain. A battery of ten cells, operating on a forty hour cycle and using a counter-current flow, must therefore have a rate of flow of steep water such that the quantity used per charge of grain will pass through the cell in two hours. Hence the minimum rate of flow of steep water in a commercial steeping battery is a hundred gallons per hour per square foot. By inspection of Figs. 6 and 7, it may be seen that increasing the rate of flow of steep water by recirculation will not materially increase the rate of diffusion of soluble materials.

It may also be seen from Figs. 6 and 7, that in a pilot plant steeping battery, using cells three to five feet deep, the steep water should be recirculated in each cell in order to simulate plant conditions. The minimum rate of flow of ten to twenty gallons per hour per square foot, gives a diffusion coefficient materially lower than that at one hundred gallons per hour per square foot.

#### Miscellaneous Observations

Figure 8 shows that for all runs performed in this

experiment, the moisture content of the grain reached 90 per cent of saturation during the first ten hours of steeping.

Judging by the odor, the sulfur dioxide was absorbed by the grain during the first three hours' steeping. This confirms the opinion of Curry and Sayre (5) that sulfur dioxide should be added with the entering grain, so that the grain will be protected from bacterial action during the entire steeping process.

A rate of flow of more than a thousand gallons per hour per square foot, up through the grain, will cause the grain to float due to turbulence. This is undesirable in a steeping vat, since the grain may be carried over into connecting pipes and pumping apparatus.

#### SUMMARY

1. A method was developed for studying the effect of rate of flow of steep water, the effect of temperature, and the effect of various chemical agents on the rate of diffusion of soluble materials from grain.

2. It was shown by experimental data that there is no advantage to be gained by recirculating steep

water in a steeping battery of the size and type used in the wet milling of corn. The data apply to the wet milling of kafir by methods similar to the wet milling of corn.

3. Steep water should be recirculated in a steeping battery of pilot plant size, in order that plant conditions may be simulated in the matter of rate of diffusion.

4. Results of the experiments show that sulfur dioxide should be added with the fresh grain, in order that the grain may be protected from bacterial action during the entire steeping process.

#### ACKNOWLEDGMENT

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